A FUNDAMENTAL STUDY OF
ROLLING CONTACT FATIGUE

By:

N.W. Kirshenbaum

Quarterly Progress Report Number 4

February 1, 1962 to May 1, 1962

Prepared under:

Contract NOw 61-0656-c
Department of the Navy
Bureau of Naval Weapons

Attention: RAPP-4

Submitted by:

ALLOYD ELECTRONICS CORPORATION
35 Cambridge Parkway
Cambridge 42, Massachusetts

Introduction

This report describes efforts made during this reporting period to prepare lithium fluoride crystals for rolling contact studies. A considerable amount of this time was spent in evaluating those methods mentioned in the previous report which seemed to promise satisfactory conditions for rolling studies. Information found in the literature corroborated our difficulties and suggested irradiating the LiF; the improvements and effects of such a treatment are described later in this report.

I. Continuation of Work Conducted to Prepare Surface of As-Received LiF for Rolling Studies.

A. Chemical Polishing and Etching

As discussed in previous reports, chemical polishing of cleaved, as-received crystals presented a severe problem in that uniform removal of the surface to expose a lower dislocation density was unsuccessful. Polishing had been attempted in aqueous solutions similar to those described by Gilman (1,2); both weakly acidic (acetic acid) and weakly basic (ammonium hydroxide) solutions were tried. Because such polishing techniques were detrimental to the crystal surface or produced no results of value, a polish using fluoboric acid (HBF_4)

B. Cleavage of Crystals at Liquid Nitrogen Temperature.

Cleaving LiF after quenching it in liquid nitrogen was investigated with the hope that the crystals would temporarily be made more brittle; this could allow the crystals to be cleaved with less damage and consequent generation of dislocations. Results were disappointing -- rather than increasing brittleness, at times the crystals seemed even more resilient, requiring several blows to fracture.

C. Annealing of Crystals Following Cleavage

Annealing procedures, also mentioned in the previous report, were studied. Cleaved crystals were annealed in a neutral atmosphere at various temperatures between 350°C and 500°C for periods of time between 1 and 18 hours. Samples were furnace-cooled to preclude deformations due to thermal stresses. Some thermal etching was observed in the crystals which had beer annealed at high temperatures for the longer periods of time. In none of these crystals was much reduction in dislocation density noted after etching.

II. Irradiation of LiF

A. Purposes

As has been described in this program's progress reports to date, it has become obvious that the LiF crystals as received from the Harshaw Chemical Company often are cleaved with some difficulty. Indeed, the cleaved faces contain a high density of dislocations, rendering them unsuitable for studies involving initiation of additional dislocations. It was mentioned in the previous report that the LiF crystals are actually slightly ductile. Lack of brittleness certainly impedes cleavage, resulting in appreciable deformation of the crystal, including cleavage on (110) planes besides on the desired (100) planes.

Difficulties which occur in using these "soft" crystals are believed to be due to their high purity; crystals formerly grown by Harshaw were unintentionally hardened by small amounts of divalent impurities (3). A paper was presented by Biletto and Cadoff (4) early this year which described strengthening of LiF by introduction of Mg into the parent lattice. Mg was introduced by precipitating MgO on the crystal surface, placing the crystal in a desiccator, and allowing Mg to

diffuse into the LiF. Interior regions of the crystal were used. Although this method presumably would result in crystal properties similar to those found in earlier Harshaw crystals, it was deemed simpler to use irradiation to harden the LiF so that it might be cleaved with minimum deformation.

A paper by Nadeau and Johnston was recently published (3) which described gamma ray irradiation of LiF to produce hard crystals which could be cleaved and handled without much damage (e.g., introduction of dislocations). Nadeau and Johnston used a 10,000-curie cobalt-60 source of gamma rays. An irradiation of four hours with a flux of 4×10^5 r/hr. produced the desired hardening.

As there is no cobalt-60 source larger than 350 curies in New England, another type of radiation was considered. Neutron irradiation of LiF has been studied by Gilman and Johnston (5); as-irradiated crystals are described as completely brittle and hardened. Whapham (6) has irradiated LiF with 1 Mev electrons but does not state what effects are produced on cleavage. A high voltage electron source (van de Graaf accelerator) was

available nearby at Massachusetts Institute of Technology. A LiF crystal in the as-received condition was irradiated from opposite sides (i.e., two passes) with 2.5 MeV electrons. The average dose was estimated at 2×10^6 rad.

B. Results of Irradiation

1. Properties of Irradiated Crystal

A slight increase in temperature was noted following irradiation. The crystal had acquired a green color which appeared emerald when viewed through the length of the crystal. This deep green color faded after one or two days; apparently relaxation produced slow room-temperature annealing. Optical absorption was investigated to determine the correspondence, if any, to findings of Nadeau and Johnston following gamma irradiation. Their study showed a strongabsorption band at 242 mμ and a weaker band at 445 mμ. Our spectrophotometer scanned only from 350 to 650 mμ and found an absorption band at 445 mμ; thus, it can be presumed that high voltage electron irradiation produced similar atomistic effects as gamma irradiation.

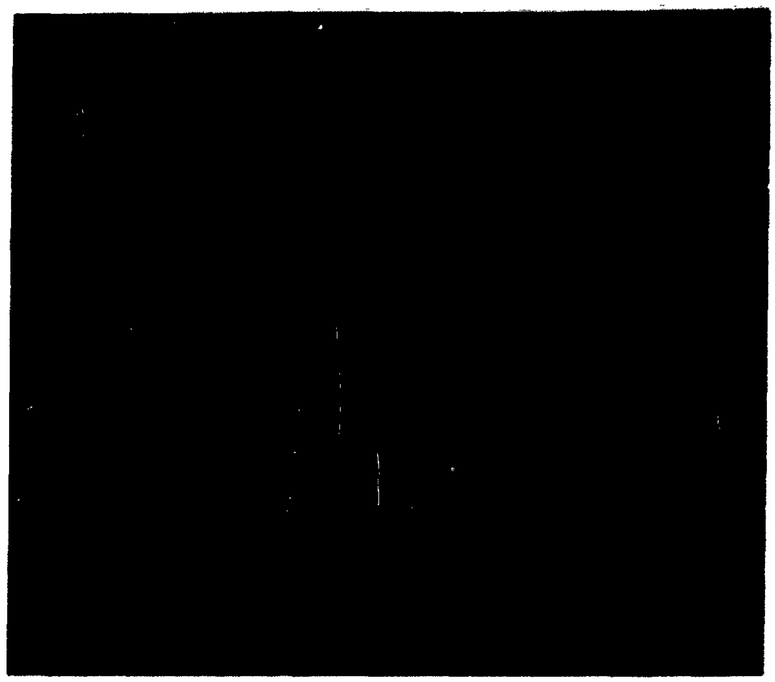
2. Effect of Irradiation upon Cleavage and Density of Dislocations.

The irradiation effects upon cleavage were striking. Crystals were cleaved with far less effort and correspondingly far less deformation than was formerly necessary. When viewed unetched at 500X magnification no significant differences were noted in the cleaved surface of the irradiated crystal; after etching, however, these surfaces were seen to be far less deformed and, consequently, freer of etch pits than non-irradiated crystal faces.

III. Rolling Studies and Planned Work

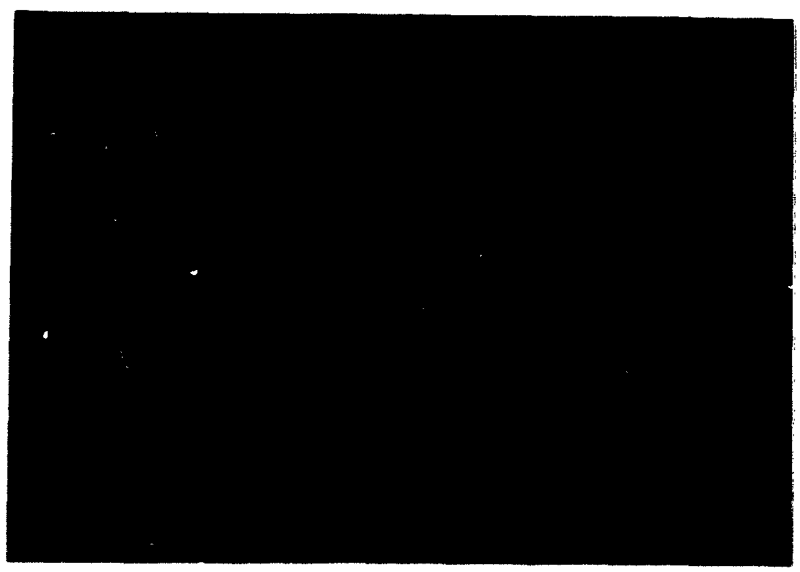
Although a not insignificant number of etch pits are still present after cleaving and etching faces of irradiated crystals, the surface is amenable for use in rolling studies. Radiation effects can be removed by annealing techniques. Nadeau and Johnston state that annealing for about two hours at 450°C removes all optical and mechanical effects of gamma irradiation, apparently returning the crystals to their original (soft) condition. Irradiated and annealed crystals will be subjected to rolling studies during the next quarter.

Figure 1



LiF etch pits revealed by 1.5×10^{-4} molar FeF_3 etchant

Figure 2



Etch pits showing dislocations induced by teflon ball rolled with 200 gram load.

REFERENCES

1. J.J. Gilman, C. Knudsen, and W.P. Walsh, J.Appl. Phys. 29, 601 (1958).

2. J.J. Gilman and W.G. Johnston, General Electric Research Laboratory Report No. 57-RL-1662, Jan.,1957.

3. J.S. Nadeau and W.G. Johnston, J.App. Phys. 32, 2563 (1961).

4. J.C. Bilello and I.B. Cadoff, Slip Activation in Magnesium Doped LiF Crystals; paper presented at AIME Annual Meeting, Feb. 21, 1962, New York.

5. J.J. Gilman and W.G. Johnston, J. Appl. Phys. 29, 877 (1958).

6. A.D. Whapham, Phil. Mag. 3, 103 (1958).